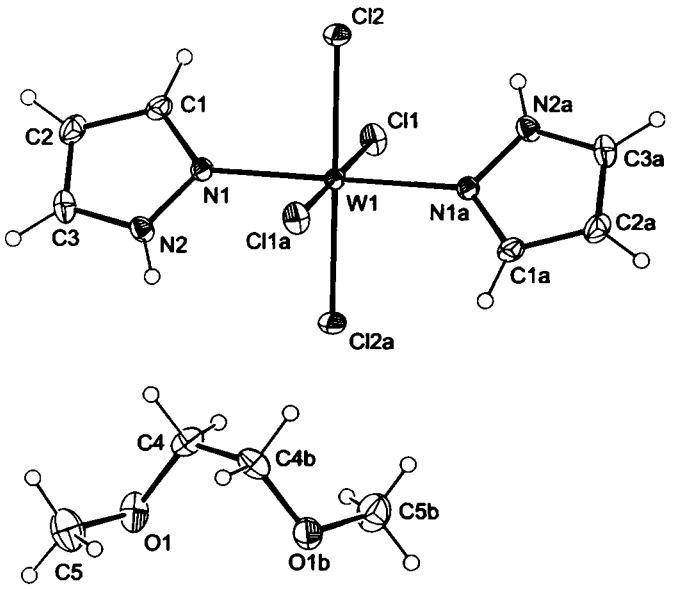
© by Oldenbourg Wissenschaftsverlag, München

# Crystal structure of tetrachloro-bis(pyrazole)tungsten(IV) dimethoxyethane solvate, $WCl_4(C_3H_4N_2)_2 \cdot (CH_3OCH_2)_2$

R. D. Ernst*, B. G. Harvey and A. M. Arif

University of Utah, Department of Chemistry, 315 S. 1400 E., Rm. 2020, Salt Lake City, Utah 84112-0850, USA

Received April 12, 2005, accepted and available on-line April 27, 2005; CCDC no. 1267/1528




## Abstract

$C_{10}H_{18}Cl_4N_4O_2W$, monoclinic, $C12/c1$ (no. 15), $a = 13.9659(3)$ Å, $b = 8.2642(2)$ Å, $c = 15.8814(3)$ Å, $\beta = 101.184(1)°$, $V = 1798.2$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.037$, $wR_{ref}(F^2) = 0.092$, $T = 200$ K.

## Source of material

The compound was synthesized from $WCl_4$(dme)/pyrazole (dme = dimethoxyethane) and crystallized by slowly cooling a concentrated solution of the complex in dme to -30 °C [1].

## Discussion

A *trans* configuration has been found for the $WCl_4(C_3N_2H_4)_2$ complex, similar to that reported for an analogous Rh(III) anion [2], but differing from the *cis* configuration exhibited by a Pt(IV) complex [3]. The complex has crystallographically imposed inversion symmetry, leading to a relatively small number of bonding parameters. The W—(Cl1, Cl2, and N1) bond distances are 2.352(1) Å, 2.344(1) Å, and 2.134(3) Å, respectively. All *trans* bond angles are required by symmetry to be linear, while the *cis* angles come in symmetry-related pairs. Thus, the Cl1–W–Cl2 angles are 89.30(4)° and 90.70(4)°, the N1–W–Cl1 angles are 89.86(9)° and 90.14(9)°, and the N1–W–Cl2 angles are 89.4(1)° and 90.6(1)°. Each tungsten complex is accompanied by one molecule of dimethoxyethane. The solvent backbone adopts a nonplanar *gauche* conformation, with a twofold rotation axis running through the center of its C–C bond. The oxygen atoms engage in hydrogen bonding interactions with the H2 atoms (on N2), leading to the formation of one-dimensional polymer chains parallel to the *a*,*c* diagonal.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | red prism, size 0.15 x 0.28 x 0.35 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 70.25 cm$^{-1}$ |
| Diffractometer, scan mode: | Nonius KappaCCD, $\varphi/\omega$ |
| $2\theta_{max}$: | 55.78° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 3514, 2128 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 1809 |
| $N(param)_{refined}$: | 99 |
| Programs: | SIR97 [4], SHELXL-97 [5] |

**Table 2.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(2A) | 8*f* | 0.1149 | 0.8097 | 0.1407 | 0.032 |
| H(1) | 8*f* | 0.1651 | 1.1333 | -0.0228 | 0.034 |
| H(2) | 8*f* | 0.0523 | 1.2556 | 0.0646 | 0.042 |
| H(3) | 8*f* | 0.0234 | 1.0409 | 0.1677 | 0.040 |
| H(4A) | 8*f* | 0.4227 | -0.0639 | 0.2690 | 0.046 |
| H(4B) | 8*f* | 0.5006 | 0.0286 | 0.3399 | 0.046 |
| H(5A) | 8*f* | 0.2853 | 0.2645 | 0.1978 | 0.084 |
| H(5B) | 8*f* | 0.2890 | 0.0710 | 0.1972 | 0.084 |
| H(5C) | 8*f* | 0.3555 | 0.1729 | 0.1448 | 0.084 |

**Table 3.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| W(1) | 4*d* | ¼ | ¾ | 0 | 0.0154(2) | 0.0172(2) | 0.0231(2) | 0.00139(6) | 0.0039(1) | 0.00026(6) |
| Cl(1) | 8*f* | 0.17330(7) | 0.8106(2) | -0.14175(6) | 0.0307(6) | 0.0384(7) | 0.0260(5) | 0.0099(5) | 0.0031(4) | 0.0020(5) |
| Cl(2) | 8*f* | 0.36836(8) | 0.9468(1) | -0.00928(7) | 0.0263(5) | 0.0259(5) | 0.0453(6) | -0.0068(4) | 0.0131(4) | -0.0020(5) |

* Correspondence author (e-mail: ernst@chem.utah.edu)

**Table 3.** Continued.

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| O(1) | 8*f* | 0.4037(2) | 0.1727(5) | 0.2702(2) | 0.033(2) | 0.057(2) | 0.040(2) | –0.003(2) | 0.008(1) | –0.023(2) |
| N(1) | 8*f* | 0.1618(2) | 0.9264(4) | 0.0470(2) | 0.018(2) | 0.021(2) | 0.029(2) | 0.002(1) | 0.006(1) | –0.001(1) |
| N(2) | 8*f* | 0.1134(2) | 0.9007(4) | 0.1117(2) | 0.025(2) | 0.029(2) | 0.027(2) | 0.007(1) | 0.009(1) | 0.006(1) |
| C(1) | 8*f* | 0.1407(3) | 1.0799(5) | 0.0217(2) | 0.032(2) | 0.019(2) | 0.034(2) | 0.002(2) | 0.007(2) | 0.004(2) |
| C(2) | 8*f* | 0.0785(3) | 1.1492(5) | 0.0696(3) | 0.030(2) | 0.024(2) | 0.050(3) | 0.006(2) | 0.007(2) | –0.004(2) |
| C(3) | 8*f* | 0.0632(3) | 1.0313(5) | 0.1257(2) | 0.027(2) | 0.040(2) | 0.033(2) | 0.008(2) | 0.008(2) | –0.009(2) |
| C(4) | 8*f* | 0.4642(3) | 0.0340(5) | 0.2800(3) | 0.042(3) | 0.038(2) | 0.035(2) | –0.012(2) | 0.006(2) | 0.001(2) |
| C(5) | 8*f* | 0.3275(4) | 0.1701(8) | 0.1968(3) | 0.046(3) | 0.072(4) | 0.045(3) | 0.010(3) | 0.002(2) | –0.011(3) |

*Acknowledgment.* We thank the University of Utah for partial support of this work.

## References

1. Harvey, B. G.: The coupling chemistry of metal pentadienyl compounds with alkynes and imines: unique C–C and C–N agostic interactions. Ph.D. Dissertation, University of Utah, USA 2005.
2. Pruchnik, F. P.; Jakimowicz, P.; Ciunik, Z.; Zakrzewika-Czerwinska, J.; Opolski, A.; Wietrzyk, J.; Wojdat, E.: Rhodium(III) complexes with polypyridyls and pyrazole and their antitumor activity. Inorg. Chim. Acta **334** (2002) 59-66.
3. Minacheva, L. Kh.; Fedotova, T. N.; Kuznetsova, G. N.: Synthesis and crystal structure of a platinum(IV) complex with pyrazole: *cis*-tetrachlorobis(pyrazole-*N*)platinum(IV), $[Pt(C_3H_4N_2)_2Cl_4]$. Zh. Neorg. Khim. **46** (2001) 240-243.
4. Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R.: SIR97 – a new tool for crystal structure determination and refinement. J. Appl. Crystallogr. **32** (1999) 115-119.
5. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.